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Measurement of Dielectric Properties of Minerals at Microwave Frequencies

By W. E. Webb and R. H. Church



UNITED STATES DEPARTMENT OF THE INTERIOR

MEASUREMENT OF DIELECTRIC PROPERTIES OF MINERALS AT MICROWAVE FREQUENCIES

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ERRATA

Page 2, equation 2: Insert equal sign (=) after Z_{in} .

Page 2, three lines above equation 5: Change "and substituting u for $e^{-2\gamma l}$;"
to "and substituting u for $e^{-2\alpha l}$;"

Page 3, equation 6: Change second minus sign in numerator to plus sign.

Page 3, equation 14 should read as follows:

$$\gamma = [RG - w^2LC + jw(GL + RC)]^{1/2}$$

Page 3, equation 15 should read as follows:

$$\gamma = jw\sqrt{LC} \left[1 + \frac{GL + RC}{jwLC} \right]^{1/2}$$

Page 4, equation 27 should read as follows:

$$\frac{R_{in}}{X_{in}} = \frac{1-u^2}{2u \sin(2\xi)},$$

Page 4, first line under equation 34: Change "this calculation"

to "these calculations"

Page 4, column 2, in line 6 of "Small-Loss Approximation" change "From equation 9"

to "From equation 12"

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UNITED STATES DEPARTMENT OF THE INTERIOR
Donald Paul Hodel, Secretary

BUREAU OF MINES
Robert C. Horton, Director

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CONTENTS

	<i>Page</i>		<i>Page</i>
Abstract	1	Test cell dimensions	5
Introduction	2	Quarter-wavelength effect	5
Measurement technique	2	Determination of dielectric constant of bulk materials	5
Theory	2	Instrumentation and procedures	5
Lossy transmission line equation	2	Test cell	7
Application of theory to dielectric-constant and loss-tangent	3	Correction for length of connector	7
Accuracy and limitations	4	Measurement procedure	7
Inherent accuracy of impedance analyzer	4	Preliminary results	8
Small-loss approximation	4	Summary	8
		References	8

ILLUSTRATIONS

1. Relative permittivity versus acetone percentage for acetone-perchloral mixtures used to calculate dielectric constant	6
2. Conductivity versus acetone percentage for acetone-perchloral mixtures used to calculate conductivity	6
3. Relative permittivity versus water percentage for water-acetone mixtures used to calculate dielectric constant	6
4. Conductivity versus water percentage for water-acetone mixtures used to calculate conductivity	6
5. Simultaneous solution for dielectric constant of quartz ($\epsilon_r = 3.9017$) compared with acetone-perchloral mixture ..	6
6. Simultaneous solution for conductivity of quartz ($\sigma = 3.046 \times 10^{-8}$) compared with acetone-perchloral mixture ..	6
7. Test cell used to measure dielectric properties of minerals	7

TABLES

1. Comparison of measured dielectric constants with published data for liquids	8
2. Comparison of measured dielectric properties with published data for minerals	8

UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

cm	centimeter	mm	millimeter
°C	degrees Celsius	MΩ/cm	megohm per centimeter
GHz	gigahertz	Ω	ohm
H/m	henry per meter	Ω/m	ohm per meter
Hz	hertz	pct	percent
in	inch	S	siemens
m	meter	vol pct	volume percent
MHz	Megahertz		

SYMBOLS USED IN THIS REPORT

ϵ_r	relative dielectric constant	L	inductance
$\tan \delta$	tangent loss factor	C	capacitance
Z	impedance	G	conductance
Γ	reflection coefficient	ω	angular frequency
γ	propagation constant	μ	permeability
R	resistance	ϵ	permittivity
ρ	resistivity	σ	conductivity
X	reactance		

MEASUREMENT OF DIELECTRIC PROPERTIES OF MINERALS AT MICROWAVE FREQUENCIES

By W. E. Webb¹ and R. H. Church²

ABSTRACT

This report describes a method the Bureau of Mines devised for measuring the dielectric constant and loss tangent of low-loss minerals at microwave frequencies. Determination of these properties makes it possible to predict the effects of using microwave heating in potential mineral processing applications. The method uses a test cell and radio-frequency (RF) impedance analyzer with automated data acquisition and analysis.

In this initial phase of microwave heating research, the Bureau measured dielectric constants and loss tangents at frequencies between 100 MHz and 1 GHz. The objective was to establish a reliable data base for use in predicting the effects of microwave heating on minerals.

Comparison with previously measured data showed the Bureau's measurement method to be accurate to within ± 3 pct. This report describes the method and discusses the preliminary results of the Bureau's investigations. Typical comparative data are included.

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INTRODUCTION

Microwave energy is widely used for industrial heating applications in the United States. The potential utilization of dielectric heating in minerals processing depends in part on the selectivity of microwave energy absorption by a given mineral and transparency of the other minerals. Absorption of microwave energy by any specific material may be determined from the electrical properties of the materials involved, namely, the dielectric constant (ϵ) and loss tangent ($\tan \delta$). The dielectric constant determines how much electrostatic energy can be stored in the mineral, and the loss tangent determines the amount of power lost as heat to the mineral. Thus the resulting heating is directly proportional to the product of these factors.

In order to predict the rate of heating of a material, it is necessary to know both its thermal and electrical properties. In particular, it is necessary to know the relative permittivity (dielectric constant) and dielectric loss (loss tangent), since the rate at which microwave power is absorbed is approximately proportional to the product of these two quantities.

In the literature, data on permittivities and loss tangents of materials important to mineral processing are either inadequate or nonexistent. Only a few references (1-4)³ give any data for minerals, and the data in these references are limited to a very small number of materials. Furthermore, much of the published data omits the frequency range from

about 30 MHz to 3 GHz. The probable reason is that, until recently, this has been a difficult range in which to work, being too high for RF techniques and too low for conventional microwave techniques. It is this frequency range which is of interest, however, because it includes the 915- and 2,450-MHz frequency bands allocated for use in microwave heating in industrial, scientific, and medical (ISM) applications.

Another problem with using published data is that minerals are not simple compounds; usually they are mixtures whose composition and crystal structure can vary widely. Samples taken from different deposits, or even from different parts of the same deposit, can have significantly different electrical properties. Other factors, such as water content, can also affect both the permittivity and loss tangent. Thus, even when published data exist, they cannot necessarily be applied indiscriminately to a given sample under investigation.

Other important areas that could benefit from an increased understanding of the electrical properties of rocks and minerals include through-the-earth communications, ground-penetrating radar, and dielectric separation of minerals.

In the initial phase of its microwave heating research, the Bureau addressed measurements of the permittivity and loss tangents at frequencies between 100 MHz and 1 GHz because of the paucity of available data in this range.

MEASUREMENT TECHNIQUE

A short section of rigid coaxial waveguide is filled with a mixture of the powdered mineral whose properties are to be measured and a liquid with a known dielectric constant and loss tangent. One end of the line is shorted and the other end connected to an RF impedance analyzer that measures the characteristic impedance looking into the line. The effective dielectric constant and loss tangent of the mineral-liquid mixture are then calculated from the input impedance and the dimensions of the transmission line. By using a series of liquids with different dielectric properties, it is possible to determine the dielectric properties of the mineral alone.

The lossy transmission line equations required for interpretation of the data are derived in the next section. Later, the accuracy and limitations of the measurement technique are discussed, and a procedure for determining the dielectric constant and loss tangent of the bulk mineral from measurements on the powdered mineral-liquid mixture is presented. Details of the experimental apparatus and measurement procedures are also given, and the preliminary results are discussed.

THEORY

Lossy Transmission Line Equation

Consider a transmission line of length l , with a characteristic impedance Z_0 , that is terminated by a short circuit. The impedance Z_{in} , looking into the line, is related to the reflection coefficient Γ (θ) by

$$\Gamma = \frac{Z_{in} - Z_0}{Z_{in} + Z_0} \quad (1)$$

Thus,

$$Z_{in} = \frac{1 + \Gamma}{1 - \Gamma} Z_0 \quad (2)$$

where Γ is related to the reflection coefficient Γ_{load} by

$$\Gamma = \Gamma_{load} e^{-2\gamma l}, \quad (3)$$

where e is the base of the natural log and γ is the complex propagation constant for the line. For an ideal short circuit, where $\Gamma_{load} = -1$,

$$Z_{in} = \frac{1 - e^{-2\gamma l}}{1 + e^{-2\gamma l}} Z_0 \quad (4)$$

Letting

$$\gamma = \alpha + j\beta, \quad (4a)$$

$$Z_{in} = R_{in} + jX_{in}, \quad (4b)$$

and

$$Z_0 = R_0 + jX_0, \quad (4c)$$

where α , β , R_{in} , jX_{in} , R_0 , and jX_0 are real and imaginary parts of γ , Z_{in} and Z_0 ; and substituting u for $e^{-2\gamma l}$; equation 4 may be separated into its real and imaginary parts to obtain the relation

$$R_{in} = \frac{(1 - u^2)R_0 - (2u \sin(2\beta l)X_0)}{1 + u^2 + (2u \cos(2\beta l))}; \quad (5)$$

³ Italic numbers in parentheses refer to items in the list of references at the end of this report.

$$X_{in} = \frac{(1-u^2)X_0 - (2u \cdot \sin(2\beta l)R_0)}{1+u^2 + (2u \cdot \cos(2\beta l))}. \quad (6)$$

The characteristic impedance and propagation constant for any transmission line can be expressed in terms of the inductance (L) per unit length, capacitance (C) per unit length, leakage conductance (G) per unit length, series resistance (R) per unit length, and angular frequency (ω) by the relations

$$Z_0 = \left(\frac{R + j\omega L}{G + j\omega C} \right)^{1/2} = R_0 + jX_0 \quad (7)$$

$$\text{and} \quad \gamma = \sqrt{(R + j\omega L)(G + j\omega C)} = \alpha + j\beta. \quad (8)$$

For a coaxial transmission line,

$$R = \frac{1}{2\pi \delta_s \sigma_c} ((1/a) + (1/b)), \quad (9)$$

$$C = \frac{2\pi\epsilon}{\ln(b/a)}, \quad (10)$$

$$G = \frac{2\pi\sigma}{\ln(b/a)}, \quad (11)$$

$$\text{and} \quad L = \frac{\mu}{2\pi} \left[\ln \frac{b}{a} + \frac{\delta_s}{2} \left(\frac{1}{a} + \frac{1}{b} \right) \right], \quad (12)$$

where a is the radius of the inner conductor; b is the inner radius of the outer conductor; μ , ϵ , and σ are the permeability, permittivity, and conductivity, respectively, of the material between the conductors; σ_c is the conductivity of the conductor; and δ_s is the skin depth in the conductor, at a frequency f :

$$\delta_s = 1/(\pi f \mu \sigma_c)^{1/2}. \quad (13)$$

Equations 10 and 11 are generally valid, while equations 9 and 12 hold only at frequencies high enough that δ_s is small compared with both the radius of the inner conductor and the wall thickness of the outer conductor. At 1 MHz, δ_s is 0.08 mm in gold. In silver and copper at 1 GHz, δ_s is less than 0.003 mm. The minimum radius of the inner conductor is >2 mm; hence $\delta_s \ll a$. For the same reason, the second and third terms in equation 12 can be ignored, as δ_s approaches zero at the higher frequencies. This is demonstrated by equation 13.

For the case of lines having small losses (i.e., $R \ll \omega L$ and $G \ll \omega C$), equation 8 may be expanded:

$$\gamma = [(\omega^2 LC + RG + j\omega(GL + RC))]^{1/2}. \quad (14)$$

Disregarding the product RG ,

$$\gamma = j\omega(LC)^{1/2} \left[1 + \frac{GL + RC}{(LC)^{1/2}} \right]^{1/2}, \quad (15)$$

using the approximation

$$(1+X)^{1/2} \approx 1 + \frac{1}{2} X, \quad (16)$$

where $X \ll 1$:

$$\gamma = \alpha + j\beta = j\omega(LC)^{1/2} + \frac{1}{2} \left[R \left(\frac{C}{L} \right)^{1/2} + G \left(\frac{L}{C} \right)^{1/2} \right]. \quad (17)$$

In a similar manner and with the additional approximation $R/L \ll G/C$, it can be shown that

$$Z_0 = R_0 + jX_0 \approx \frac{(L/C)^{1/2}}{(1 + \tan^2 \delta)^{1/2}} + j \frac{1}{2} \frac{(L/C)^{1/2} \tan \delta}{(1 + \tan^2 \delta)^{1/2}}, \quad (18)$$

$$\text{where} \quad \tan \delta = \frac{\sigma}{\omega \epsilon} \quad (19)$$

is the loss tangent for the dielectric filling the line.

Application of Theory to Dielectric-Constant and Loss-Tangent Measurement

If it is assumed that the resistance per unit length of the transmission line (R) is known or can be measured, then R_0 , X_0 , β , and u can be expressed in terms of σ and ϵ using equations 4, 9 through 12, and 17 through 19. These expressions could then be substituted into equations 5 and 6 to obtain a pair of simultaneous equations for σ and ϵ that could, in principle, be solved to obtain the permittivity and conductivity (or loss tangent) from the measured values of the input impedance $R_{in} + jX_{in}$. The resulting equations are intractable even using numerical methods. An iterative method of determining the dielectric constant and loss tangent from the input impedance is therefore used.

Substituting C and L from equations 10 and 12 into equation 17, disregarding the second term in equation 12, and assuming $\mu = \mu_0$,

$$\beta = 2\pi f (\mu \epsilon)^{1/2} = 2\pi f (\mu_0 \epsilon_0 \epsilon_r)^{1/2}; \quad (20)$$

$$\beta = \frac{2\pi f}{C} (\epsilon_r)^{1/2}. \quad (21)$$

If a lossless line is assumed as an initial approximation, then $u=1$ and $X_0=0$, and equation 6 reduces to

$$X_{in} = \frac{\sin(2\beta l) R_0}{1 + \cos(2\beta l)} = R_0 \tan(\beta l). \quad (22)$$

However,

$$R_0 = \left(\frac{L}{C} \right)^{1/2} = \frac{\ln b/a}{2\pi} \left(\frac{\mu_0}{\epsilon_r \epsilon_0} \right)^{1/2} \quad (23)$$

$$R_0 = \frac{R_0(\text{air})}{(\epsilon_r)^{1/2}}, \quad (24)$$

and

$$\frac{2\pi f l}{C} \cdot \frac{X_{in}}{R_0(\text{air})} = \frac{\tan \xi}{\xi}, \quad (25)$$

where ξ is a substitution for

$$\xi = \frac{2\pi fl}{C} (\epsilon_r)^{1/2}. \quad (26)$$

Equation 25 can be solved numerically for ξ , and ϵ_r can then be obtained from equation 26.

Taking the ratio of equations 5 and 6,

$$\frac{X_{in}}{R_{in}} = \frac{1 - u^2}{2u \sin(2\xi)}, \quad (27)$$

$$\text{and} \quad u^2 + \left(2 \frac{R_{in}}{X_{in}} \sin(2\xi)\right)u - 1 = 0, \quad (28)$$

which can be solved for u using the quadratic formula to obtain

$$u = -k + (k^2 + 1)^{1/2}, \quad (29)$$

$$\text{with} \quad k = \frac{X_{in}}{R_{in}} \sin(2\xi). \quad (30)$$

Once the value for u is obtained, α is found from

$$\alpha = -\frac{1}{2l} \ln u; \quad (31)$$

but from equations 17, 23, and 24,

$$\alpha = \frac{1}{2} R \frac{(\epsilon_r)^{1/2}}{R_{0(air)}} + \frac{1}{2} [GR_{0(air)}] / [(\epsilon_r)^{1/2}]. \quad (32)$$

Using equation 11,

$$\alpha = \frac{R(\epsilon_r)^{1/2}}{2R_{0(air)}} + \frac{\pi \sigma R_{0(air)}}{1n(b/a)(\epsilon_r)^{1/2}} = -\frac{1}{2l} \ln u; \quad (33)$$

hence,

$$\sigma = \frac{-1n(b/a)(\epsilon_r)^{1/2}}{\pi R_{0(air)}} \cdot \left[\frac{R(\epsilon_r)^{1/2}}{2R_{0(air)}} + \frac{1}{2l} \ln u \right]. \quad (34)$$

The values of ϵ_r and σ obtained from this calculation are substituted into the following two expressions obtained from equation 18:

$$R_0 = \frac{R_{0(air)}}{(\epsilon_r)^{1/2}(1 + \tan^2 \delta)^{1/2}} \quad (35)$$

$$X_0 = \frac{R_{0(air)} \tan \delta}{2(\epsilon_r)^{1/2}(1 + \tan^2 \delta)^{1/2}} \quad (36)$$

These values, along with the previous value of u , are substituted into equation 6, which is solved numerically for a new value for ϵ_r . This value is then used to compute a new value of u from the ratio of equations 5 and 6:

$$\begin{aligned} (R_0 - \frac{R_{in}}{X_{in}} X_0) u^2 + 2(X_0 + \frac{R_{in}}{X_{in}} R_0) \sin(2\beta l) u \\ - (R_0 - \frac{R_{in}}{X_{in}} X_0) = 0 \end{aligned} \quad (37)$$

This process is continued until consistent values of ϵ_r and σ are obtained. The loss tangent, $\tan \delta$, may then be computed from the conductivity, σ , using equation 19.

ACCURACY AND LIMITATIONS

Inherent Accuracy of Impedance Analyzer

According to the manufacturer's specifications the impedance analyzer used for these tests has an accuracy of 1 pct. Thus it is desirable to keep other sources of error below this value. Errors resulting from approximations in the analysis, dimensional tolerance of the test cell, etc., are discussed in the following paragraphs.

Small-Loss Approximation

In the derivation of the previous section, the assumptions made were that $R \ll \omega L$ and $G \ll \omega C$. These assumptions will now be examined to see what limitations they impose on the materials that can be measured using the technique described. First consider the assumption that $R \ll \omega L$. From equation 9, the inductance per unit length of a 50- Ω coaxial line is found to be 1.67×10^{-7} H/m. Thus ωL is approximately 10 Ω /m at 10 MHz, or 1,000 Ω /m at 1 GHz. The resistance per unit length computed from equation 9, using the conductivity of gold, is found to be approximately 0.08 Ω /m at 10 MHz, or 0.8 Ω /m at 1 GHz. Consequently, R is from two to three orders of magnitude smaller than ωL over the frequency range of interest.

For the second assumption, $G \ll \omega C$, note that since $G/C = \sigma/\epsilon$,

$$\frac{\sigma}{\omega \epsilon} = \tan \delta \ll 1 \quad (38)$$

$$\text{or} \quad \delta \ll 2\pi f \epsilon_0 \epsilon_r. \quad (39)$$

One further assumption implied in the derivation is that

$$\frac{R}{Z_0} + GZ_0 \ll \beta. \quad (40)$$

Substituting typical values for the transmission lines leads to the requirement that the resistivity, ρ , of the sample meets the following conditions:

$$\begin{aligned} \rho &>> 160 \text{ at } 100 \text{ MHz;} \\ \rho &>> 16 \text{ at } 1 \text{ GHz} \end{aligned} \quad (41)$$

Most minerals listed in standard tables of dielectric properties such as those compiled by Von Hippel (8) and Parkhomenko (1) easily meet the requirements of equations 38, 39, and 41. The notable exceptions are a few coals and some conductive ores, such as the metallic sulfides. Thus it is concluded that the equations used to compute the dielectric constant at room temperature are sufficiently accurate for most minerals.

Test Cell Dimensions

The characteristic impedance (Z_0) of the empty test cell is determined by the ratio of the radii of the outer and inner conductors (b and a, respectively) through the relation

$$Z_0 = 60 \ln(b/a). \quad (42)$$

Because of the complex way in which Z_0 enters into calculation of ϵ_r and $\tan \delta$, it is not possible to determine analytically the effect of errors in the cell diameter. Therefore, data on a sample of pure acetone were used to calculate ϵ and $\tan \delta$ assuming values of Z_0 from 49 to 51 Ω . It was found that an error in Z_0 of 0.5 Ω led to an error of 0.25 pct in ϵ and 0.69 pct in $\tan \delta$. Thus an error of $\pm 0.5 \Omega$ in Z_0 appears to be acceptable. By differentiating equation 42 and allowing the error in a and b to contribute equally to the error in Z_0 , it was found that the tolerance for the inner and outer diameter would be ± 0.002 in for a and ± 0.004 in for b.

Because of the small diameter of the inner conductor, it was impossible to maintain the required tolerance. Fortunately, the line impedance depends only on the ratio b/a. It was therefore possible to select an inner conductor close to the desired size and then ream the outer conductor to give the correct ratio.

Analysis of the error introduced by uncertainty in the length of the cell (l) indicated that the percent error in both ϵ and $\tan \delta$ is approximately twice the percent error in l . Thus, if the accuracy in measuring l for a 10-cm-long cell was ± 1 mm, an error of ± 2 pct in the computed values of ϵ and $\tan \delta$ would result.

Quarter-Wavelength Effect

If the length of the test cell is an odd multiple of one-fourth of the guide wavelength in the cell, a voltage null exists at the input port of the cell. This results in a low signal-to-noise ratio at the test port of the impedance analyzer and hence a decrease in accuracy. In fact, at frequencies sufficiently close to the frequency that produces the quarter-wavelength condition, the analyzer output becomes unstable and varies randomly. Clearly these frequencies must be avoided.

In order to cover the entire frequency range from 1 MHz to 1 GHz, two or more test cells of different lengths are used. For each cell, the data for which $0.22 n\lambda_g < l < 0.28 n\lambda_g$ are discarded, where λ_g =guide wavelength, l =length of cell, and n =an odd integer. In this way the entire frequency range may be covered.

DETERMINATION OF DIELECTRIC CONSTANT OF BULK MATERIALS

At higher microwave frequencies, the usual techniques for dielectric measurements involve cutting a block of material so that it just fills a portion of the rectangular waveguide. In the case of the measurements described here, this was impractical for several reasons; namely, the presence of the center conductor, the small size of the transmission line, and the difficulty of machining mineral samples. Measurements were therefore made with powdered samples. Measurements using powders, however, do not yield the dielectric properties of the bulk solid but that of a solid-air mixture. A number of investigators have proposed models for relating the dielectric constant of a mixture to the dielectric constants of its component (β).

An alternate technique for determining the dielectric constant of the bulk material was developed. This technique is based on the assumption that if a material is immersed in a liquid having the same dielectric constant, then the dielectric constant of the mixture will be the same as that of the liquid.

The test procedure is based on the premise that if the test cell is filled with a finely powdered sample of the material whose dielectric constant is to be measured, and a liquid of known dielectric constant is then added until all the space between the solid particles is filled, the dielectric constant of the mixture can then be determined. This procedure is repeated using liquids with different dielectric constants until the dielectric constant of the mixture is the same as that of the liquid alone.

In order to be able to continuously vary the dielectric constant of the liquid, mixtures of two liquids are used. For minerals with dielectric constants between 2.3 and 20.6, a mixture of perchloral (tetrachloroethylene) and acetone is used; for dielectric constants between 20.6 and 75.63, a mixture of acetone and deionized water is used. The dielectric constants of these mixtures were measured and found to be an almost linear function of the percentage of the higher dielectric component. Using a least-mean-square technique, a quadratic curve was fit to the data.

Figure 1 is the fitted plot of the dielectric constant (referred to as relative permittivity on the plot) of the acetone-perchloral (liquid-liquid) mixtures. Figure 2 is the fitted plot of the conductivity of these mixtures. From these data the dielectric constant and tangent loss can be calculated from the previously derived equations for the resistance (R) and reactance (X) values measured with the impedance analyzer. The dielectric properties can be determined for minerals with dielectric constants between 2.3 and 20.6 and conductivities between 1×10^{-5} and 2×10^{-4} S. Similarly, the data used to derive figures 3 and 4 can be used to calculate the dielectric properties for minerals with dielectric constants between 20.6 and 76.6 and conductivities between 2×10^{-4} and 2×10^{-3} S at 900 MHz. The dielectric constant and conductivity can be computed for any desired frequency between 100 MHz and 1 GHz, with the corresponding tangent loss calculated from the conductivity value.

The dielectric properties of a mineral are determined by conducting a series of 11 tests with the mineral in the test cell. Each test is conducted by immersing the mineral in a liquid-liquid mixture whose dielectric properties are known. The dielectric constant (ϵ_r) and conductivity (σ) for the mineral and liquid-liquid mixture are measured in each test. This experiment is then repeated with varying percentages of each liquid-liquid mixture. A curve can then be plotted from the data points of the mineral and liquid-liquid mixture combinations. The intercept point of this curve with the liquid-liquid mixture curves (figs. 1-4) is where the dielectric constant (for quartz, figure 5) and the conductivity (for quartz, figure 6) are equal. From the conductivity value, the loss tangent is calculated from equation 19. This calculation holds true for small values of $\tan \delta < 1$.

INSTRUMENTATION AND PROCEDURES

The coaxial test cell is connected to a Hewlett Packard (HP) 4191A⁴ RF impedance analyzer. This analyzer applies an RF signal to the input of the test cell and measures the magnitude and phase of the applied and reflected voltage by

⁴ Reference to specific products does not imply endorsement by the Bureau of Mines.

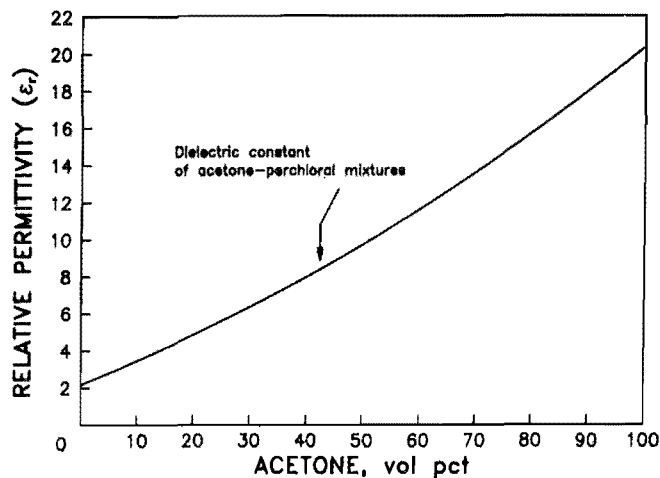


FIGURE 1.—Relative permittivity versus acetone percentage for acetone-perchloral mixtures used to calculate dielectric constant.

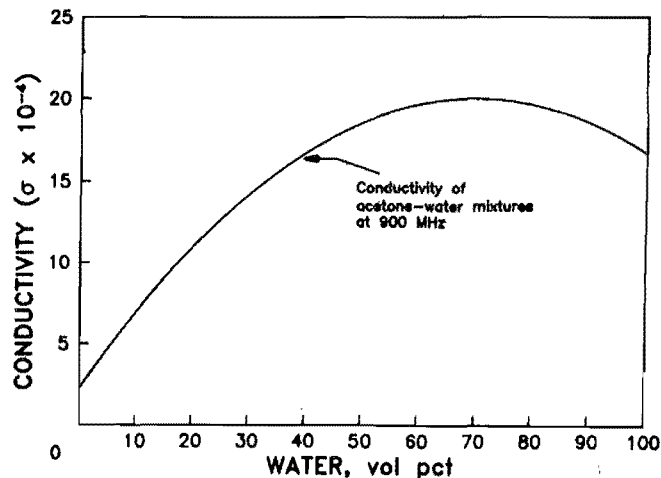


FIGURE 4.—Conductivity versus water percentage for water-acetone mixtures used to calculate conductivity.

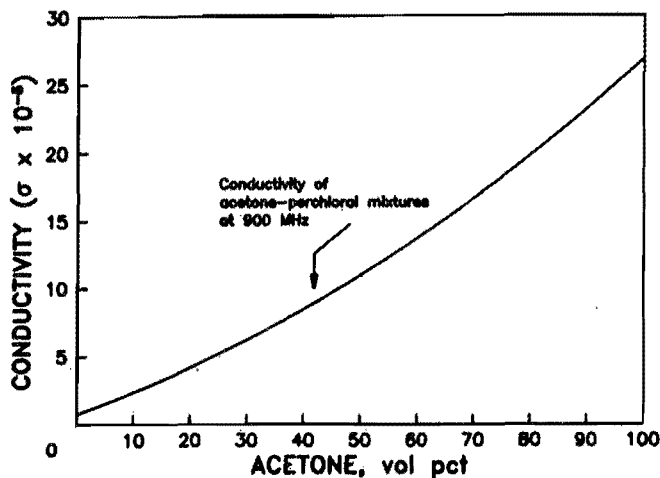


FIGURE 2.—Conductivity versus acetone percentage for acetone-perchloral mixtures used to calculate conductivity.

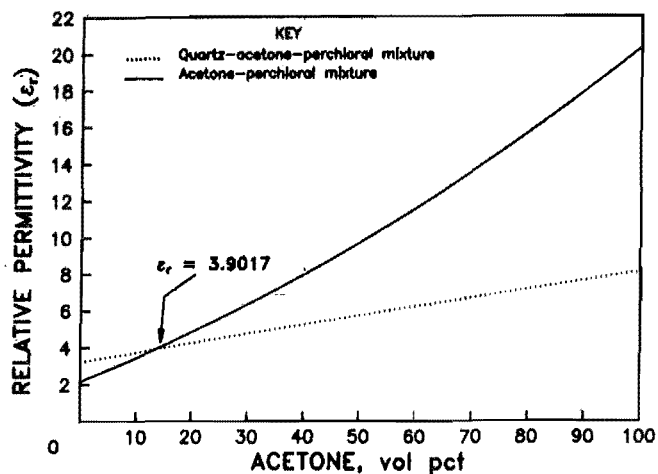


FIGURE 5.—Simultaneous solution for dielectric constant of quartz ($\epsilon_r = 3.9017$) compared with acetone-perchloral mixture.

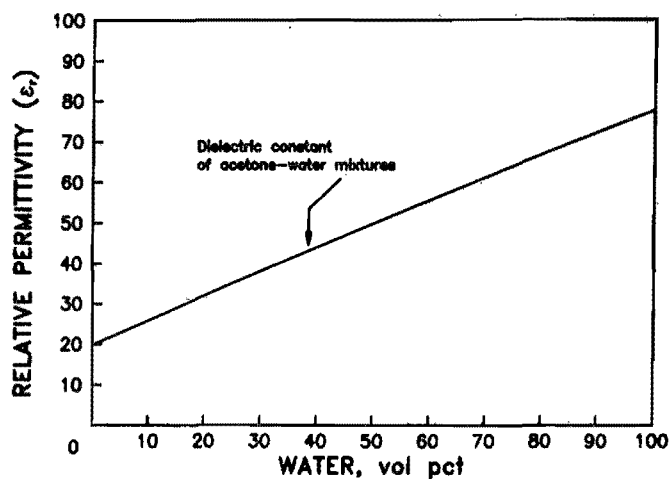


FIGURE 3.—Relative permittivity versus water percentage for water-acetone mixtures used to calculate dielectric constant.

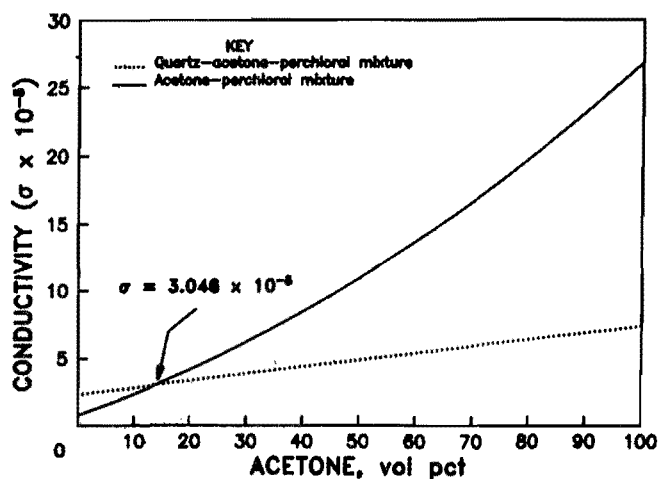


FIGURE 6.—Simultaneous solution for conductivity of quartz ($\sigma = 3.046 \times 10^{-9}$) compared with acetone-perchloral mixture.

means of a directional bridge. Comparison of these voltages gives the complex reflection coefficient at the input of the test cell. This reflection coefficient can be displayed either as real and imaginary parts or as magnitude and phase. In addition, a microprocessor within the HP-4191A analyzer can compute the input impedance of the line from the reflection coefficient. The frequency of the signal can be varied from 1 MHz to 1 GHz in steps as small as 100 Hz.

The impedance analyzer is controlled by a HP-1000 computer. The computer is programmed to transmit the control signals necessary to sweep the analyzer frequency and record the input impedance at each frequency step. Data from the analyzer are transmitted to the computer, where they are analyzed to determine the dielectric constant and loss tangent of the material in the test cell.

Test Cell

A test cell is shown in figure 7. The base of the test cell is mounted on a 7-mm connector which attaches to the device-under-test (DUT) port of the impedance analyzer. The top of the cell is shorted with a screw-on cap. A set screw on the cap insures good electrical contact between the cap and inner conductor. The cells are designed so that they may be completely disassembled for cleaning.

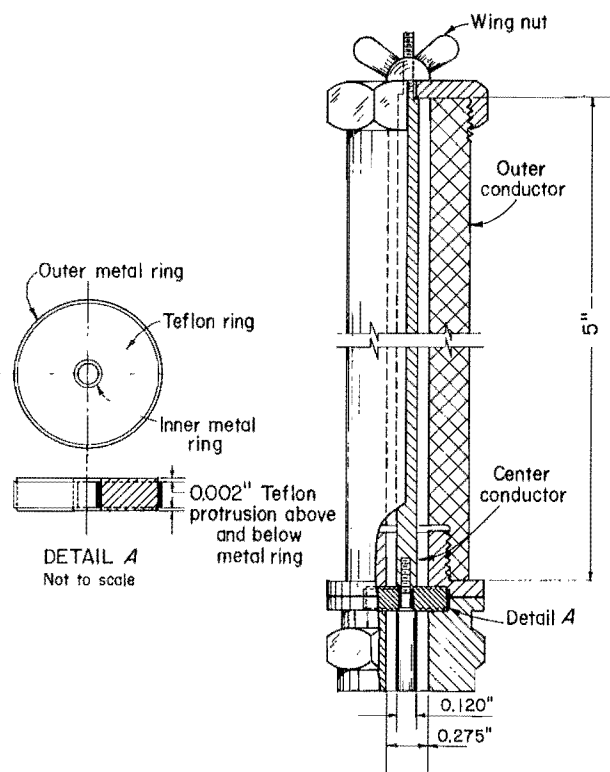


FIGURE 7.—Test cell used to measure dielectric properties of minerals.

The inner and outer conductors of the test cell are constructed of brass, as is the shorting cap. A Teflon fluorocarbon polymer washer is used to seal the base of the cell. The diameters of the conductors are chosen to be the same as the corresponding dimensions of the 7-mm connector.

Correction for Length of Connector

The input impedances used in the calculations are assumed to be measured at the input end of the test cell. However, the impedance analyzer measures the impedance at its own output port. These two points are separated by the length of the 7-mm connector and the thickness of the Teflon washer. The impedance analyzer has provisions for compensating for this separation provided that the electrical length is known. The electrical length is defined as the distance in air that would give the same phase shift as the line connecting the device under test to the impedance analyzer. To measure the electrical length, the outer conductor is removed from the base of the test cell and replaced by a shorting plug. The reflection coefficient is then measured and the electrical length, l_e , computed from the relation

$$l_e = -\frac{1}{2\beta} \ln \Gamma_{in} \quad (43)$$

Measurement Procedure

Before making a measurement, the test cell is cleaned with dilute hydrochloric acid to remove any oxidation from the surfaces of the conductors that would increase their electrical resistance. The cell is then filled with the mineral sample and perchloral. The cell is then connected to the impedance analyzer, and the program that controls the analyzer is initiated. Under computer control, the frequency is swept from 100 MHz to 1 GHz, usually in 20-MHz steps. The impedance is measured at each frequency and stored in the computer. The computer reads the frequency, resistance, and reactance. The dielectric constant and loss tangent are then computed at each frequency and stored. This process is repeated for the acetone-perchloral mixture from 0 to 100 pct acetone in 10-pct steps. After the final run, the dielectric constant of the solid is computed. For high-dielectric (>20) materials, the procedure is the same except that water-acetone mixtures are used.

All data collection and processing is automated and is done under computer control. The computer program (written in FORTRAN) controls the operation of the impedance analyzer. The program collects the data (frequency, resistance, and reactance), converts the data from character form to real numbers, and stores the data on a hard disk for later retrieval. The program makes corrections to compensate for the various electrical lengths of the individual test cells. A subroutine is used in the main program to calculate the dielectric constant (epsilon) and conductivity (sigma) at each frequency from the resistance and reactance of the mineral using the previously derived equations.

PRELIMINARY RESULTS

In order to verify the accuracy of the measurement technique, the dielectric constants of a number of materials were measured and compared with published data. In particular, standard samples of nitrobenzene, cyclohexane, and dichloroethane obtained from the National Bureau of Standards (NBS) were measured. Other samples included reagent-grade acetone and perchloral, as shown in table 1.

The variation from the accepted value for the dielectric constant is approximately 3 pct, as predicted by the error analysis presented in the "Accuracy and Limitations" section. Table 2 shows the results for a typical low-loss mineral, quartz. The measured dielectric constant differs from the published value by 1.3 pct and the measured loss tangent by 2.5 pct.

These results and measurements on a number of other minerals indicate that the measurement technique is accurate for liquids and solids with low to moderate conductivity. Measurements on materials with very high conductivity have been less satisfactory. The principal problem encountered with very lossy materials is that the wave traveling through the coaxial test cell is attenuated to such a degree that the signal-to-noise ratio at the input of the impedance analyzer is degraded to the point where measurement of the input impedance becomes impossible. Fortunately, introduction of a

lossy material into the test cell also reduces the guide wavelength, allowing the use of a shorter test cell. It is therefore believed that by using shorter test cells the measurement technique can be extended to conductive minerals. Other methods are also being investigated to measure these high-loss minerals.

TABLE 1.—Comparison of measured dielectric constants with published data for liquids.

Liquid	Dielectric constant (ϵ_r)		Difference, pct
	Measured value	Reference value	
Cyclohexane ¹	² 1.953	³ 2.01517	3.10
1,2-Dichloroethane ¹ ..	² 10.14	³ 10.3551	2.07
Nitrobenzene ¹	² 33.72	³ 34.7416	2.94
Perchloral ⁴	² 2.27	⁶ 2.30	1.30
Acetone ⁴	² 20.53	⁶ 20.70	.80
Water ⁷	⁵ 75.63	⁶ 78.54	3.70

¹ Obtained from NBS; dielectric constant reference material.

² Averaged from 3 measurements.

³ NBS value at 25°C.

⁴ Reagent-grade material.

⁵ Averaged from 2 measurements.

⁶ Maryott and Smith (5).

⁷ Deionized water at 18 MΩ/cm.

TABLE 2.—Comparison of measured dielectric properties with published data for minerals

Mineral	Dielectric constant (ϵ_r)			Loss tangent ($\tan \delta$)		
	Measured value	Reference value ¹	Difference, pct	Measured value	Reference value ¹	Difference, pct
Quartz	3.90	3.85	1.3	1.56×10^{-4}	1.6×10^{-4}	2.5
Corundum	9.1	9.34	2.57	1.30×10^{-4}	NA	NA
Calcite	8.87	8.6	3.14	5.56×10^{-5}	NA	NA
Muscovite	4.05	5.4	25.00	1.5×10^{-4}	NA	NA
Periclase	9.41	9.65	2.49	4.52×10^{-5}	NA	NA
Cerrusite	22.65	18.6	22.8	4.57×10^{-4}	NA	NA

NA Not available.

¹ All from Young (2).

SUMMARY

The Bureau of Mines has devised a technique for measuring the dielectric constants and loss tangents of minerals in the frequency range from 100 MHz to 1 GHz. The experimental apparatus necessary to implement this technique has been constructed and tested. Data acquisition and analysis is fully automated, requiring only loading of the test cell, con-

necting the test cell to the analyzer, and starting the computer program.

Experimental measurements on previously measured materials verify that this technique is accurate to about 3 pct for minerals with low to moderate conductivity, i.e., dielectric constants from 2.3 to 76.6 and loss tangents from 1×10^{-5} to 2×10^{-3} .

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